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THE EFFECT OF PARTICLE SIZE ON THE CHARACTERIZATION OF ACTIVATED CARBON FROM TROPICAL BLACK BAMBOO (*Gigantochloa atroviolacea*)

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ABSTRACT

Activated carbon also known as activated charcoal is a common term for carbon materials, which comprises charcoal. Activated carbon has a good adsorption capacity against gases and pollutants in liquids because of its wide surface. The material used in this research was tropical black bamboo, with the variation of particle size -10+20 mesh, -20+40 mesh, -40+60 mesh and -60+80 mesh. There are 2 processes to produce activated carbon, i.e. carbonation and activation. In this research, the carbonation is set at 380°C in 1 hour. Then, each size of carbon was activated by H₃PO₄ 9.8% along 24 hours. The analysis conducted were moisture content, ash content, iodine number, and functional group analysis using Fourier Transform Infrared Spectroscopy (FTIR). The smaller particle size, the more pores will be produced and it caused the surface area higher. The results showed that variation of particle size had effect on the characterization and quality of activated carbon, where moisture content is between 10.60 to 4.05%, ash content is 1% to 0.2% and iodine adsorption is between 710 mg/g to 900 mg/g. In FTIR analysis, it showed that all of the samples had O-H, C=C, and C-O functional group.

INTISARI**Kata Kunci:**

Karbon aktif
Limbah bambu
Karbonasi
Aktivasi
Asam Fosfat

Karbon aktif atau disebut juga arang aktif adalah istilah umum untuk bahan karbon, yang terdiri dari arang. Karbon aktif memiliki kapasitas adsorpsi yang baik terhadap gas dan polutan dalam cairan karena permukaannya yang luas. Material yang digunakan dalam penelitian ini adalah limbah bambu wulung yang berasal dari industri kerajinan tirai bambu. Variasi ukuran partikel pada penelitian ini adalah -10+20 mesh, -20+40 mesh, -40+60 mesh, dan -60+80 mesh. Terdapat 2 proses untuk menghasilkan karbon aktif, yaitu karbonasi dan aktivasi. Pada penelitian ini karbonasi diatur pada suhu 380°C selama satu jam. Kemudian masing-masing ukuran karbon diaktivasi dengan H₃PO₄ 9,8% selama 24 jam. Analisis yang dilakukan adalah analisis kadar air, kadar abu, daya serap iodin, dan analisis gugus fungsi menggunakan Fourier Transform Infrared Spectroscopy (FTIR). Semakin kecil ukuran partikel maka akan semakin banyak pori yang dihasilkan dan menyebabkan luas permukaan semakin tinggi. Hasil penelitian menunjukkan bahwa variasi ukuran partikel berpengaruh terhadap karakterisasi dan kualitas karbon aktif, dimana kadar air antara 10,60 - 4,05%, kadar abu 1% - 0,2% dan adsorpsi bilangan iodium antara 710 mg / g sampai 900 mg / g. Hasil analisis FTIR menunjukkan bahwa semua sampel memiliki gugus fungsi O-H, C = C, dan C-O.

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1. INTRODUCTION

Activated carbon is an amorphous compound produced from materials containing high carbon, through a carbonation process. Activated carbon has a good adsorption capacity for gases and pollutants in liquids because of its wide surface. Activated carbon is currently very intensively used as an adsorbent, because of its high adsorption capacity and is thermally stable and easy to obtain. The raw materials of activated carbon production is from agricultural waste, coal, coconut shells, bamboo, and others [1]. Activated carbon contains hydrogen and oxygen which are attached to functional groups such as carboxyls, phenols, and ethers [2]. The fine structure of activated carbon increased the surface area ($>1000 \text{ m}^2/\text{g}$) of pores that make it has high adsorptive properties. Activated carbons are applied in various shapes, such as powder, granular, pellet, honeycomb and activated carbon fiber (ACF), and each type has its specific application [3]. Activated carbon also becomes important filter material for the removal of hazardous components in exhaust gases for the purification of drinking water and for wastewater treatment [4,5]. Carbon as adsorbent can absorb colours and odours because the contaminants and/or pollutants are bound to the active and free sites on the carbon surface until it is filled up [6].

As a type of natural plant material, bamboo is a potential renewable biomass with the advantages of fast growth and high yield. Bamboo charcoal is a new porous material with high porosity and a special micropore structure, which bamboo can obtain in high temperature and anoxic conditions. [7]. Bamboo grows fast and can be harvested every four years. Replacing wood with bamboo for the preparation of porous charcoal can reduce carbon dioxide emissions.[8]

Carbonation and activation are the process to produce activated carbon. Carbonation process aims to evaporate volatile substances, so they can open the pores of the activated carbon surface. Carbonation occurs with several stage which includes the removal of water or dehydration, a change in ingredient organics into carbon elements and tar decomposition resulting in pores carbon gets bigger. The purpose of the activation process is to increase or enlarge the pores diameter of carbon and expand the volume absorbed in the pore and to open new pores [9].

The activated carbon with good quality should comply with quality standard of activated carbon according to SNI 06-3730-1995 about standard quality and activated carbon testing, that shows in the Table 1.

Table 1. Standard of Activated Carbon SNI 06-3730-1995

Type of testing	Parameter
Moisture content	Max .15%
Ash content	Max. 10%
Iodine absorption	Min. 750 mg/g

The relevant research was done, and produced activated carbon from string bamboo (*Gigantochloa apus*) using H_3PO_4 as activator with the variation of activator concentration, time and temperature. The result of this research was the bamboo can be made into activated charcoal with an activator H_3PO_4 . Another research showed iodine absorption increased by the mesh size. From the size -20+40 mesh; -40+60; -60+80; -80+100 mesh resulted iodine adsorption by the number 228.42; 304.56; 507.60; 710.64 mg/g [10,11].

To reduce the amount of bamboo waste from handicraft industry, it requires processing bamboo waste into valuable products such as activated carbon. Based on the background, this research aims to know the effect of particle size on the characterization of activated carbon from bamboo waste using activator 9.8% H_3PO_4 . The main material is tropical black bamboo, or wulung in Indonesia, as bamboo waste from traditional curtain industry.

2. METHODS

The materials used in this research are bamboo waste, aquadest, 1% amylum or starch, 9.8% H_3PO_4 , Iodine 0.1 M and $\text{Na}_2\text{S}_2\text{O}_3$ 0.1 M. And the tools for this research are screen (size 10+20 mesh, -20+40 mesh, -40+60 mesh and -60+80 mesh), hotplate magnetic stirrer, oven, furnace, desicator, beaker glass, erlenmeyer, analytical balance.

There were the steps how the research conducted:

1. Sample preparation from bamboo waste

The sample are cut in $\pm 2 \text{ cm}$. Then wash with water and dry it at temperature 100°C until the weight is constant.

2. Carbonation

Then sample is dried at temperature 380°C around one hour using the furnace. The carbon formed then grind using mortar and sieve in various size of screen size 10+20 mesh, -20+40 mesh, -40+60 mesh and -60+80 mesh.

3. Activation

In activation step, solve 20 grams of each size of particles in the activator solution in beaker glass. The solution is 9.8% H_3PO_4 in 100 ml aquadest. Then carbonized charcoal was activated by immersing it for 24 hours. The yield of carbon is filtered and rinsed with distilled water until the pH is neutral, then put it in an oven with a temperature of 100°C for an hour. The result is cooled in decicator to room temperature.

4. Analysis

1. Water Content

Weighed 1 gram of sample in a porcelain cup and then put it in the oven at 110 °C until a constant weight is obtained. Water content is calculated by the following equation:

$$\%WC = \frac{a-b}{a} \times 100\% \dots \dots \dots (1)$$

a: initial mass

b: mass after drying

2. Ash Content

1 gram of sample in porcelain cup is heated in a furnace at 650 °C for 2.5 hours, until ash is formed, cooled in a desiccator, weighed.

$$\%Ash = \frac{\text{mass of ash}}{\text{mass of carbon}} \times 100\% \dots \dots \dots (3)$$

3. Iodine Adsorption

Weighed 0.5 grams of charcoal and then put it in the erlenmeyer, added 20 mL of 0.10 N iodine solution, stirred for 15 minutes at room temperature. Then the mixture is centrifuged until two layers are formed. 8 mL of supernatant pipette, put into Erlenmeyer. The solution is titrated with sodium thiosulfate solution using the starch indicator 1%, until the blue color disappears.

4. Functional Group Analysis

The functional groups of activated carbon were determined using FTIR method, conducted in Laboratorium Terpadu UII. The spectrum results are then compared with literature.

3. RESULT AND DISCUSSION

3.1. Result

Based on the characterization of activated carbon as adsorbent from bamboo waste by the variation of particle size are -10+20 mesh (F1), -20+40 mesh (F2), -40+60 mesh (F3), -60+80 mesh (F4) with activator 9.8% H_3PO_4 , the activated carbon that formed is powder and black. Then the activated carbon has been analyzed by moisture content, ash content, iodine number and FTIR. The following table shows the result of the analysis:

Table 2. Data of moisture content, ash content and iodine number

Parameter	Sample F1	Sample F2	Sample F3	Sample F4
Moisture content	10.60%	9.34%	7.16%	4.05%
Ash content	1.0%	0.9%	0.8%	0.2%
Iodine number (mg/g)	710.64	808.98	837.54	900.99

Table 3. Data of FTIR analysis of each sample

	Wavenumber (cm ⁻¹)	Functional group	Intensity
F1	3466.69	O-H	13.682
	1595.57	C=C	23.657
	1229.26	C-O	27.151
F2	3425.41	O-H	28.707
	1596.85	C=C	39.317

	1217.30	C-O	41.926
	3439.37	O-H	39.717
F3	1598.91	C=C	50.335
	1171.48	C-O	52.454
	3445.57	O-H	39.599
F4	1598.81	C=C	49.418
	1171.76	C-O	51.204

3.2. Discussion

3.2.1 Moisture and Ash Content

Calculation of moisture content aims to determine the hygroscopic properties of activated carbon, where generally activated carbon has a very large affinity for water. This very hygroscopic property causes the activated carbon can be used as adsorbent [12].

The moisture content analysed by heating the activated carbon at 110°C. Based on the result in Figure 1, the moisture content is decline following the particle size. When the particle size is small, it will produce small pores that cause the surface area higher. When the number of pores increases, the easier it is to dry. The sequence result of moisture content is 10.60%, 9.34%, 7.14% and 4.05%. All the percent moisture is under the maximum value of SNI, that is 15%. This result is related to the research with the variation of particle size is 100 mesh, 140 mesh and 200 mesh and produce moisture content 1.4%, 1.27% and 1.1% [13].

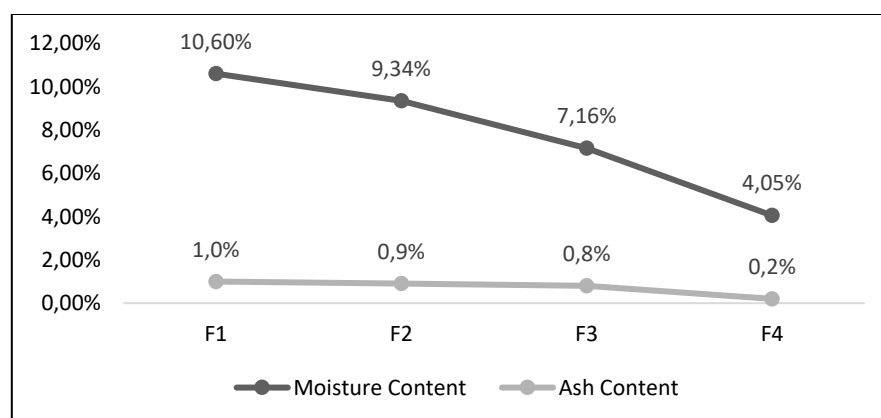


Figure 1. Moisture and ash content as a function of particle size

The analysis of ash content done by heating the activated carbon in furnace at 650°C for 2.5 hours. The result obtained based on the variation of particle size is 1%, 0.9%, 0.8% and 0.2%, respectively. When the particle size is smaller, it will produce less ash. It means that the particles with smaller sizes when heated to 650°C allow these particles to change phase into gas so the amount of ash becomes less. All the sample has low ash content, where the maximum value of ash content from SNI is 10%. Low ash content indicates that mineral oxides such as Na, K, Mg and Ca are left in very little amount of activated carbon. The presence of excess ash can cause blockage of activated carbon, so the carbon surface becomes less [13,14].

3.2.2 Iodine Number Adsorption

The purpose of this analysis is to determine the adsorption ability of activated carbon in coloured solution. The sample was analysed by iodometric method where the iodine number obtained will show the ability of its adsorption power. The efficiency of the adsorption process depends on the physicochemical characteristics of the adsorbate and adsorbent. For the latter, factors such as the pore size, area, and surface chemistry are relevant if the process is carried out in activated carbon. Adsorption capacity also depends on the characteristics of the activated carbon such as carbon content, ash content, moisture content, surface area and the yield. The main factor that greatly influences the adsorption power is the surface area of activated carbon because the adsorption mechanism is related to the number of pores [15,16].

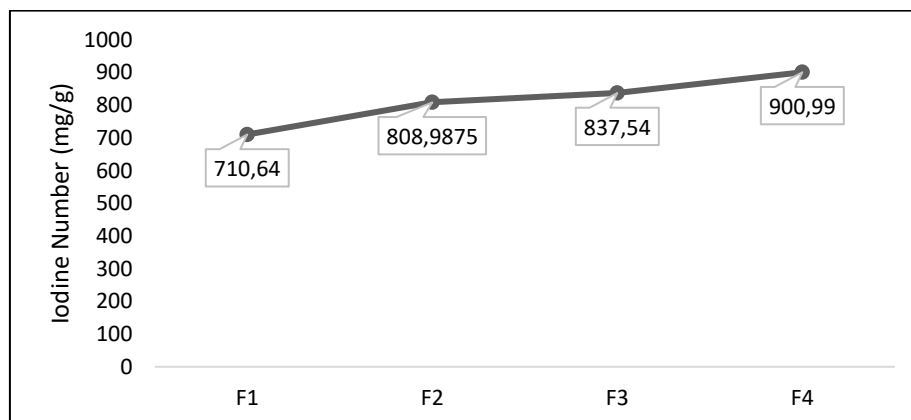


Figure 2. Iodine number adsorption as a function of particle size

From the result of this study, the iodine number increase by the smaller size of particle. The best iodine number is 900.99 mg/g from sample F4, with the size of particle is -60+80 mesh or 0.25-0.177 mm. From 4 samples, sample F1 is 710.64 mg/g, which is not achieved to SNI where the minimal iodine value is 750 mg/g. The higher the mesh value, the more pores of activated carbon open and forming a cavity that is bigger in size than the iodine molecule so that the iodine molecule enters the activated carbon [17].

3.2.3 Functional Group Analysis

The functional groups contained in activated carbon from black bamboo can be determined by FTIR Spectrophotometer. The analysis conducted on activated samples using 9.8 % H_3PO_4 . It was carried out from wave numbers 4000-400 cm^{-1} , where in the range of wave numbers there are functional groups and fingerprints that are owned by activated carbon. The results of FTIR spectra for each sample are shown in the Figure 3.

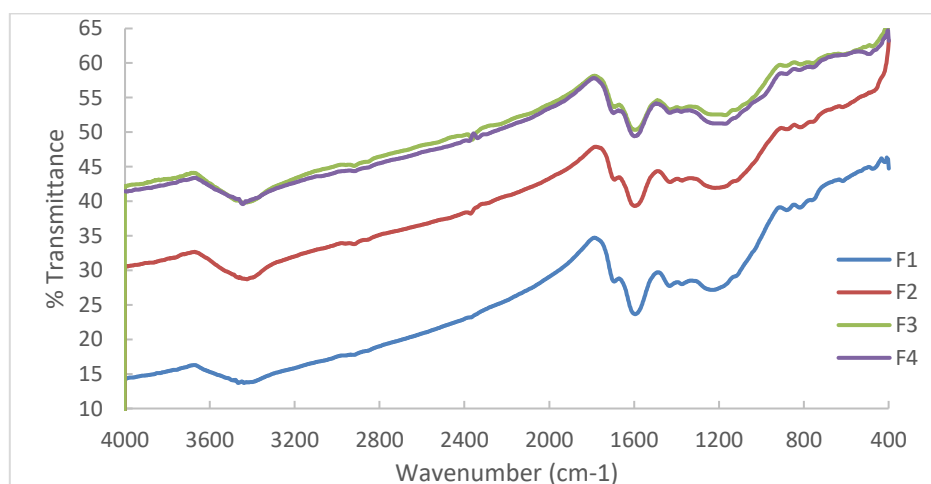


Figure 3. Spectra of FTIR analysis in bamboo activated carbon

The result analysis using FTIR showed that there were peaks appeared. Based on the wavenumber detected, all the sample are dominated with O-H, C=C and C-O group. The result was d interpret from FTIR Spectroscope of Organic Material [18]. It is proved by the absorption peaks at 3466.69 (F1), 3425.41 (F2), 3439.37 (F3), and 3445.57 (F4) for O-H stretching where the number 3500-3200 cm^{-1} refer to hydroxyl group. The decrease in the absorption peak stated in the results occurs due to the decomposition of the adsorbed hydroxyl groups and water. The amount of hydroxyl groups showed a strong hydrogen bond (from carboxyl, phenol or alcohol). For wavenumber 1615–1450, it indicates C=C group (aromatic ring stretch). As showed in the figure there are absorption peaks at 1595.57 (F1), 1596.85 (F2), 1598.91 (F3) and 1598.81 (F4). And the fact that showed there is C-O group or phenol is absorption peaks at 1229.26 (F1), 1217.30 (F2), 1171.48 (F3) and 1171.76 (F4).

The presence of O-H and C-O bonds indicates that the activated carbon produced tends to be more polar. Thus, the activated carbon produced can be used as adsorbent in polar substances, tend to purify water, sugar, alcohol, or as an absorber of formaldehyde emissions [19]. Other application show that the use of activated bamboo charcoal as an adsorbent medium can reduce manganese and nitrate ions in water. The results of the study were the optimum adsorption of nitrate is 74.57% and manganese ion is 60.60% [20].

4. CONCLUSION

The waste of tropical black bamboo can be used as a prospective material in the preparation of activated carbon. This study showed that the phosphoric acid developed and increased the physical properties of activated carbon prepared from black bamboo. Based on the result analysis, it can be concluded that the variation of particle size affected the characterization and quality of activated carbon. Activated carbon with screen size - 60+80 mesh has the best quality between the other samples with low moisture content and ash content at 4.05% and 0.2% respectively. It also has high adsorption ability on iodine solution 900.99 mg/g. Activated carbon from bamboo consists of O-H, C-O, and C=C functional groups. The two oxygen-containing functional groups are responsible for the decreasing of polar substances in water and the application of this activated carbon can be expanded in future study. In example, application in purifying water or reducing hazardous metal content in waste water. This study also can be used as reference to the next study by adding the variation of time activation, so the best activation time will be known.

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